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14. ABSTRACT

Substantial work has progressed on membrane processing for improved durability. Over the quarter, cell voltage was improved by over 150 mV, while durability was improved from less than 150 hours of testing to passing the accelerated stress testing protocol. The project is generally on track for a March completion. Results are described in more detail in the following sections.

15. SUBJECT TERMS

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Quarterly Progress Report

Project Title: Improved Round Trip Efficiency for Air Independent Regenerative Fuel Cell Systems

Project Period: June 18, 2010 to June 17, 2011
Date of Report: January 27th, 2011

Recipient: Proton Energy Systems
Award Number: N00014-10-C-0369

Working Partners: W. L. Gore
Cost-Sharing Partners: None

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Project Objective:
The purpose of this effort is to investigate advanced membrane materials that enable higher efficiency electrolysis, substantially improving the practical energy density for regenerative fuel cell applications. Additionally, exercisable options in this project will advance the understanding, implementation, and operational testing of the features that enable an RFC to simultaneously be truly air independent and have high energy density.

Objectives:

- Define the key membrane attributes that correlate with performance characteristics important for device function such as proton conductivity, ion exchange capacity, nitrogen and water permeation, and visual evaluation of mechanical strength of the membrane in the seal areas of the cell. (Gore and Proton)
- Determine the optimal processing parameters (pretreatment, pressing temperature, time) of these membranes for MEA fabrication. (Proton)
- Define thickness of the membrane required to withstand sealing loads and electrical loads as well as differential pressure. (Proton)
- Define the practical performance limits of these new membranes in terms of operating current, pressure, and temperature. (Proton)
- Based on screening of membrane samples, test a refined list of potential candidates at full-sized MEA scale.

Background:
Navy underwater vehicle platforms (UUV, ASDS, SWCS, etc.) are demanding larger and larger energy storage capacities to accommodate longer underwater missions and increased platform power requirements. New energy storage devices with high volumetric energy density for underwater vehicles, both manned and unmanned, are therefore needed, such as regenerative fuel cell (RFC) systems based on proton exchange membrane (PEM) technology. An RFC consists of a fuel cell powerplant, an electrolysis system for recharging the reactants, and reactant storage. These water-based energy storage systems have been shown to perform substantially better than traditional battery systems in areas such as rechargeability, specific energy density, and reliability. Advanced membrane and catalyst materials will enable higher efficiency electrolysis, substantially improving the practical energy density for regenerative fuel cell applications.

From a full proposal to develop an advanced demonstration system, Task 5 was selected for initial study. This task focused on membrane development. The research objectives for Phase 1 of this task were broken into the following separate subtasks:

Task 5.0: Thinner, Reinforced Membranes:
Task 5.1 Alternative Membrane Material Procurement
The contractor shall procure up to four advanced alternate membrane materials from not more than two membrane suppliers. These samples will be large enough for testing at the 0.03 ft^2 cell size.

Task 5.2 Alternative Material Screening Tests
The contractor shall evaluate the advanced alternate membrane materials for strength, fluid permeation, and ionic conductivity using typical Proton procedures. Fluid permeation may be conducted at up to two temperatures and three pressures.

Task 5.3 Material Treatment Process Optimization
The contractor shall conduct not more than three process trials with each of two alternative membrane candidates.

Task 5.4 Catalyst Application Process Optimization
The contractor shall conduct not more than three process trials with each of two alternative membrane candidates.

Task 5.5 Feasibility Testing
The contractor shall evaluate the samples generated from tasks 5.4 using standard Proton procedures for lateral and cross-cell resistance measurements. The best of the three trials for each candidate shall be selected for use in integrated operational testing. Not more than 3 single-cell tests shall be supported for up to 100 hours.

Status:
Substantial work has progressed on membrane processing for improved durability. Over the quarter, cell voltage was improved by over 150 mV, while durability was

improved from less than 150 hours of testing to passing the accelerated stress testing protocol. The project is generally on track for a March completion. Results are described in more detail in the following sections.

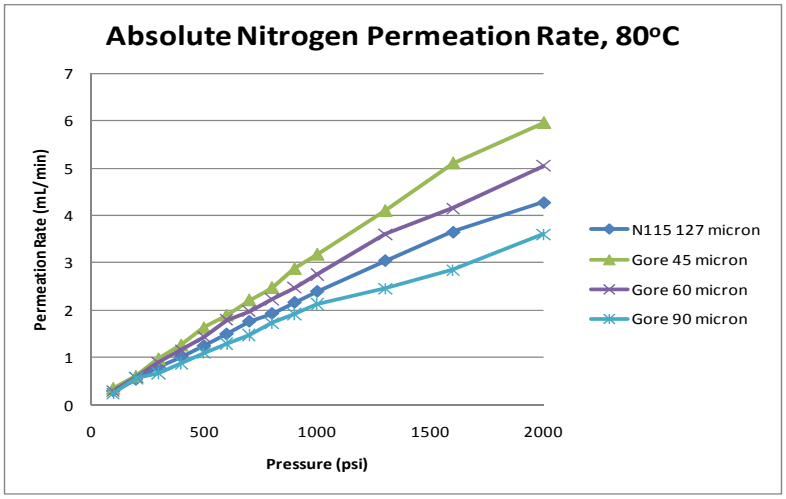
Task 5.0 Program Update: Thinner, Reinforced Membranes

Task 5.1 Alternative Membrane Material Procurement

Procurement of alternative membrane samples has been completed. The initial order from WL Gore is complete and several samples of membranes and ½ MEAs are ready for processing studies. Catalysts, baseline membrane material, and cell stack parts are all in house and ready for use.

Subtask 5.2 Alternative Material Screening Tests

Physical characterization of the baseline material has been completed. The Gore materials performed very well on diffusion tests as shown in the figure below, implying that hydrogen crossover will be comparable or lower than the baseline 5-mil Nafion material, even for membranes ½ the thickness. Ionic conductivity (as reported by WL Gore) was also within expected parameters for the various membrane samples.



Subtask 5.3 Material Treatment Process Optimization

Electrodes are fabricated using 2 methods at Proton: a Teflon-bonded approach and an inked approach. Previous studies have shown that equivalent performance can be achieved with either method, though the Teflon-bonded approach is most established. Both methods were explored under this task.

Studies were performed at several different pressing temperatures, dwell times, and humidity control. Samples were either pressed in a sealed box, which required significant ramp up time and exposure to elevated temperature, or with pre-heated platens, which allowed escape of water from the membrane but much shorter overall pressing time. On visual inspection for discoloration and deformation, as well as measurement of dimensional change of the sample, processing limits were established for decal attachment studies under Subtask 5.4.

Under wet pressing conditions within the press box, temperatures of 290-360F appeared to be acceptable. Under dry pressing conditions or with pre-heated platens, severe damage was observed on the membranes when the standard backing materials were utilized, including observations of discoloration of the membrane and severe peeling. It is believed that the membrane interaction with the backing material caused this effect, rather than temperature instability of the membrane itself. Adding a barrier layer significantly improved membrane visual characteristics. This need for a barrier layer may limit the electrode approach to the inked formulations.

Subtask 5.4 Catalyst Application Process Optimization

Initial attempts to press Teflon-bonded electrodes using the sealed box approach but within the milder processing conditions defined in Subtask 5.3 resulted in poor catalyst transfer in pressing trials with fully humidified membranes (see photo below).

In addition, the durability of these membranes was poor (see below Subtask 5.5 description). On consultation with WL Gore, the use of wet processing was not recommended. While we still believe acceptable conditions can be found, a dry inked-based approach will be explored first. Attachment of inked electrodes was carried out using various masking and barrier layer techniques designed to achieve the proper loading on the electrode for effective transfer, while protecting the membrane from exposure to the backing layer. A successful transfer with good catalyst uniformity was achieved and the samples were processed for electrochemical characterization.

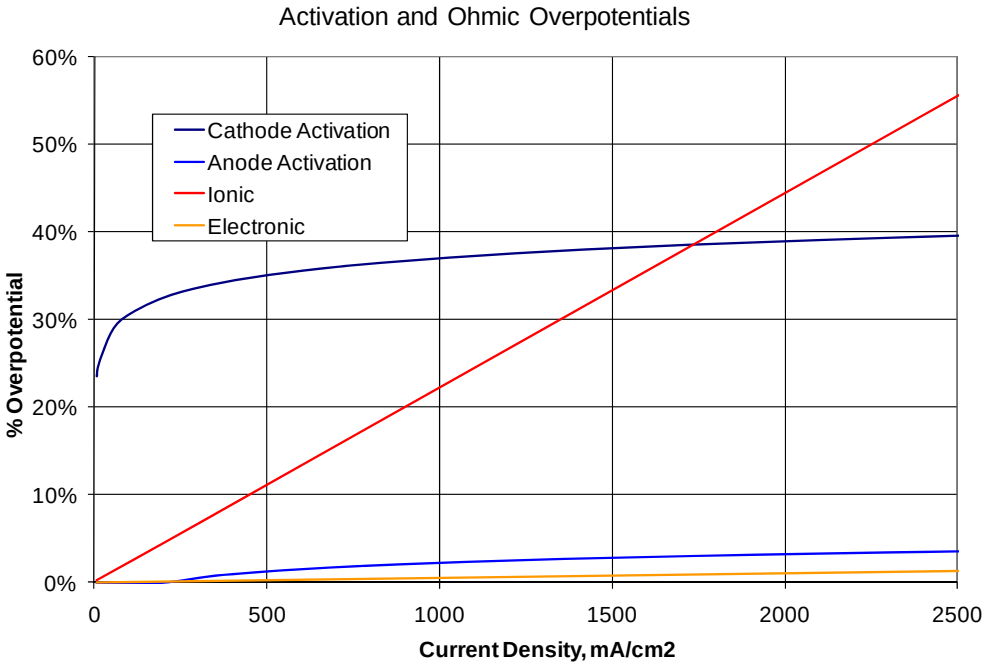
Subtask 5.5 Feasibility Testing

Wet-pressed membranes were tested in single cell tests as listed in the table below. Due to the non-uniform catalyst distribution, electrode performance was relatively poor, as demonstrated by the voltages at full current (tests 1 and 2). Typical cell stack voltages for Proton's 10-mil Nafion samples are in the 2.10-2.15V range at these current densities. In addition, tests 1 and 2 failed within the first 150 hours of electrolysis. In order to test whether the Gore samples had the same stability as earlier samples, and to see the performance of the Gore ½ MEAs to see if any insights could be gained on the source of the high potential on earlier tested Gore-manufactured MEAs, two ½

MEAs were laminated together. As seen for test 3, performance was so poor that the current had to be reduced by more than half to keep the voltage at reasonable potentials, indicating a high likelihood that the original problem was in the Gore Pt electrode rather than the anode.

Test	*Membrane Thickness*	*Anode Electrode*	*Cathode Electrode*	*Press Load (psi)*	*Press Temp ©*	*Dwell Time*	*Current Density (Amps/cm^2)*	*Cell Potential (V)*
1	90	Proton applied	Proton applied	831	102	5 minutes	1.86	2.18
2	90	Proton applied	Proton applied	831	102	5 minutes	1.86	2.26
3	90	1/2 Gore MEA (45 micron Pt/C)	1/2 Gore MEA (45 micron Pt/C)	N/A	N/A	N/A	0.7	2.33

This result was somewhat surprising in that Pt is a very good cathode catalyst and has low overpotential, as shown in Proton's electrochemical modeling for our own electrode performance below. However, this behavior is typical of other commercial samples tested by Proton and is believed to be due to the fact that these electrodes are optimized for fuel cell performance, where hydrophobic surfaces are desired and removal of gas bubbles from the surface is not a concern.



Despite the high voltage exhibited by the Gore sample, stability was improved and this sample survived the initial break-in period that the Proton electrodes had failed. This cell successfully based the accelerated stress testing of multiple pressure cycles. This

result indicates that the Gore membranes do have good stability and that the failures to date can be assigned to the processing conditions. It also shows that the Gore membranes can withstand relatively high potentials.

In the next series of tests, the inked samples described above were evaluated. One Pt-Pt MEA was fabricated for comparison with the 2 Gore ½ MEAs. The other two samples were Proton's standard catalysts, applied with the new techniques, on both the 90 micron and 60 micron samples. The 90 micron samples both successfully passed the accelerated stress testing protocol. The 60 micron sample is still pending cycling tests.

Performance results are summarized in the table below. The Pt-Pt MEA had elevated potential vs. the standard catalyst configuration, but was considerably better than the Gore MEA, being able to withstand standard operating currents. This result again supports the Pt electrode as the source of the high voltage in the Gore MEAs. The standard catalyst on 90-micron material exhibited voltages close to that expected for 7-mil Nafion, a significant improvement vs. the previous trials while maintaining durability. The 60-micron sample is the best performing to date.

Test	*Membrane Thickness*	*Anode Electrode*	*Cathode Electrode*	*Current Density (Amps/cm^2)*	*Cell Potential (V)*
4	90	Proton applied (Pt)	Proton applied	1.86	2.45
5	90	Proton applied	Proton applied	1.86	2.05
6	60	Proton applied	Proton applied	1.86	1.95

Task 5.0 Project Management and Reporting
The Principal Investigator attended the ONR S&T Conference in November. Technical reports on processing studies and electrochemical results are being drafted. A visit with WL Gore at Proton's site is scheduled for February 3.

Plans for Next Quarter and Key Issues:
In the next quarter, additional work will be performed to increase electrochemical performance of the Gore membranes with Proton catalysts and processing methods. Additional work will be performed on analysis of variables at the 60 micron level, and durability will be tested at the 45 micron level. It is anticipated work will be substantially completed for this phase, in anticipation of continued optimization and scale up in Phase 2.

Patents: None to date.

Publications / Presentations:
No new presentations during the reporting period.

Task Schedule

Task Number	Project Milestones	Task Completion Date				Progress Notes
		Original Planned	Revised Planned	Actual	Percent Complete	
1	Alternative Membrane Procurement	08/31/10	10/29/10	10/29/10	100%	Completed
2	Alternative Membrane Screening	09/30/10	2/15/11		90%	Diffusion and thickness measurements completed.
3	Membrane Treatment Optimization	12/31/10	12/21/10		100%	Range of press temperatures and durations determined
4	Catalyst Application Optimization	02/28/11			75%	Inked electrodes show improved performance
5	Feasibility Testing	06/17/11	03/31/11		50%	Several single cell stacks tested and progress shown in durability and efficiency
6	Project Management	06/17/11	03/31/11		60%	

Budget Summary

Quarter	From	To	Estimated Billing	Actual Billing
1Q10	06/18/10	9/30/10	$30,190	$34,992
2Q10	10/01/10	12/31/10	$17,500	$25,723
3Q10	1/1/11	3/31/11	$15,824	
4Q10	4/1/11	6/30/11	$13,424	
		Totals=	**$76,938**	**$60,715**